THE CHARACTERIZATION OF THE REVERSION STRESS FOR NiTi

Carliss Robert Such

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THESIS

THE CHARACTERIZATION OF THE
REVERSION STRESS FOR NiTi

by

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The Characterization of the Reversion Stress for NiTi

by

Carliss Robert Such
Lieutenant, United States Navy
B.S., Northrop Institute of Technology, 1968

Submitted in partial fulfillment of the
requirements for the degree of

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ABSTRACT

The reversion stress of NiTi is characterized under several unique thermomechanical situations. The effect of partial reversion of initial strain on the reversion stress is studied. The final stress is apparently not dependent on whether the initial strain is lost by stress-free reversion or stress-reversion. The effect of developed stress on the temperature range for the reversion transformation to austenite is also studied; A_s and A_f are observed to increase with increasing developed stress. The reversion transformation once started continues to proceed smoothly regardless of the stress. A model for the mechanism of constant-stress reversion is proposed. The effect of cooling below M_d on the retained stress and the stability of the retained stress when held at constant temperature above A_f are also discussed.

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I. INTRODUCTION

At the Naval Ordnance Laboratory, in 1961, W. J. Buehler and R. C. Wiley investigated the properties of various alloy combinations of nickel and titanium. In near-equiatomic alloy compositions of NiTi, unique properties were discovered. Specimens of this alloy exhibited low yield-stress and large plastic strains in certain temperature ranges. When heated these specimens returned to their original shape. The generic name given to the near-equiatomic alloys of NiTi is NITINOL (an acronym for Nickel Titanium Naval Ordnance Laboratory).

The ability to return to the original shape has been called the shape memory effect. The strain history of the alloy and temperature range take significant roles in the process. To display shape memory effect, an object of the proper alloy composition must first be cooled below a definable temperature. It is then strained to a new "permanent" shape. To recover the initial shape, the object must be heated above an equally definable temperature. In this process, complicated shapes may be recovered by an infinite combination of internal stresses generated within the NiTi structure. (Figure 1 illustrates this process.)

The temperatures which control the recovery process are the transformation temperatures of martensite to austenite. Also necessary for the recovery process is temperature control

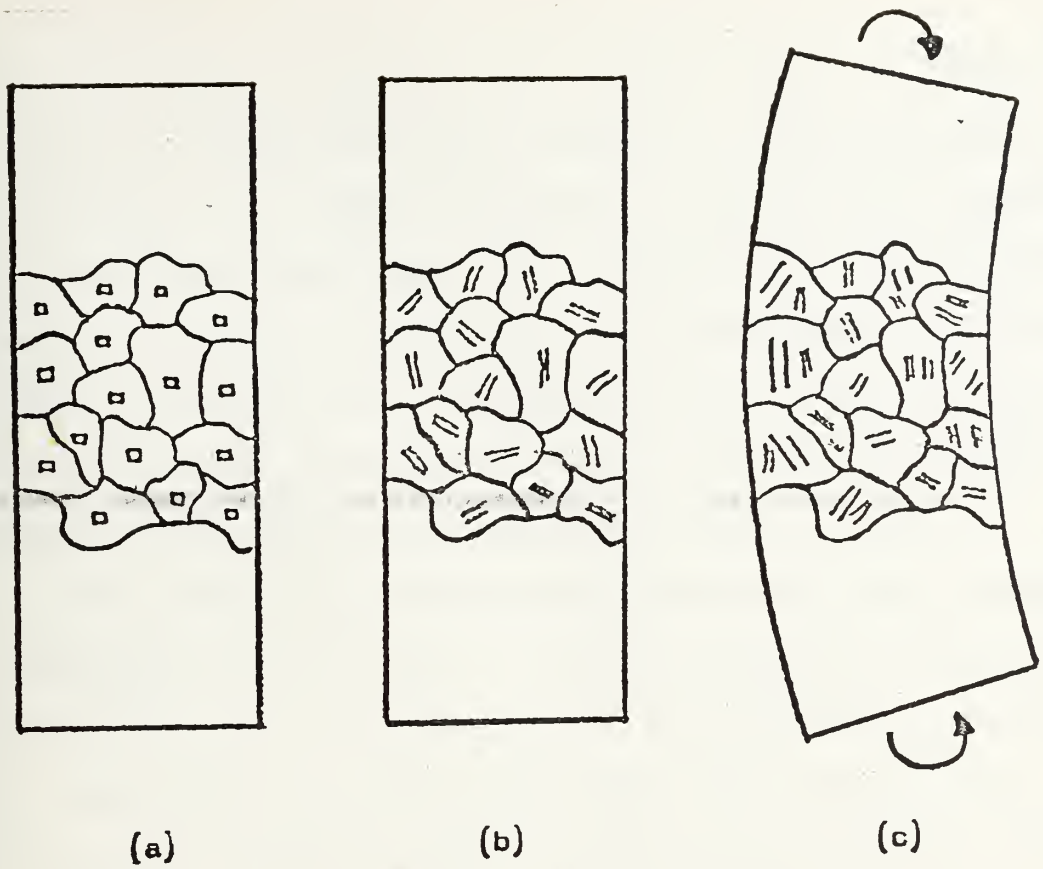


FIGURE 1. Schematic representation of the formation of deformation martensite from thermal martensite.

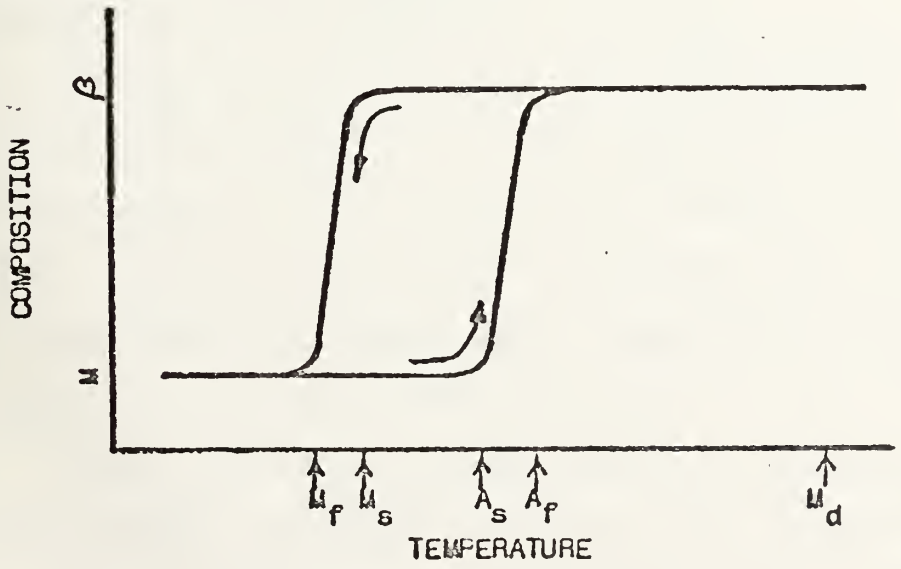


FIGURE 2. The thermal hysteresis curve for the stress-free transformations of thermal martensite and austenite.

for the initial deformation. In this case, the transformation temperatures for austenite to martensite are significant. As seen in Figure 2, NiTi exhibits a narrow thermal hysteresis loop. This phenomenon allows the shape memory effect to operate within a narrow temperature range. Shown in Figure 2 is a temperature point defined as M_d. This represents the high temperature limit for stress-induced deformation martensite formation.

The creation of deformation martensite from thermal martensite below the M_f, or from thermal austenite below the M_d, or from a combination of both between M_s and M_f, is the vehicle by which shape-memory can occur. The mechanism through which the martensitic structure is capable of shape-memory has not been fully explained (Ref. 1). Although this is an area of great interest, it was not investigated in this study.

The $A_s - A_f$ and $M_s - M_f$ temperature ranges are a function of the alloy composition. This temperature band can be varied between $-10^{\circ}C$ and $100^{\circ}C$ by changes in composition. With the partial substitution of nickel with cobalt, iron or aluminum, the transformation-temperature range may be shifted to as low as $-196^{\circ}C$ (Ref. 2).

There are three general areas for the mechanical employment of NiTi. As shown in Figure 3 (A), shape-memory effect can be used for non-structural members where shape recovery provides simplified deployment of stored appendages. In this application, only internal stresses are encountered and the member is allowed to regain its original shape.

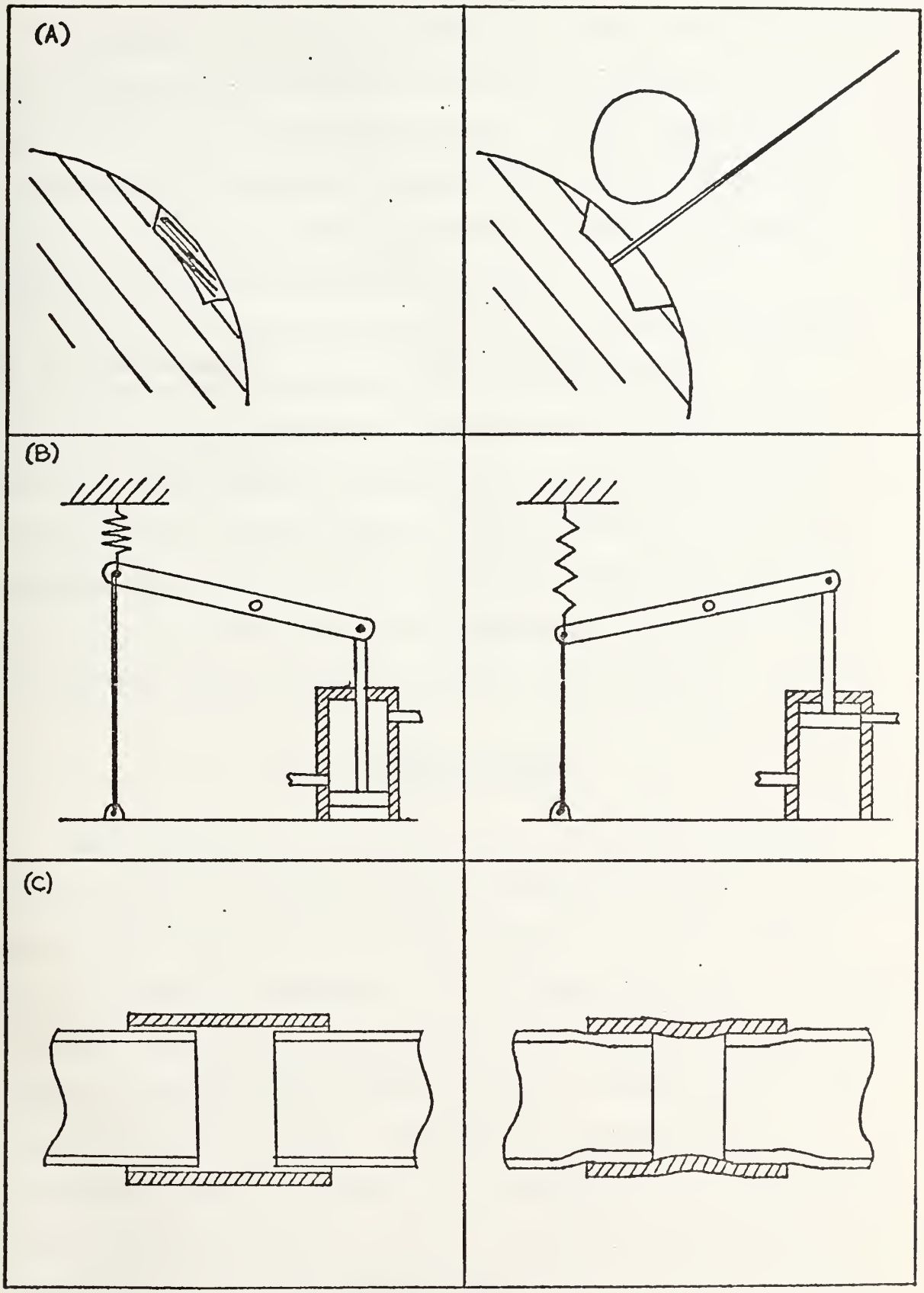


FIGURE 3. Selected applications of the shape-memory effect: (A) shape recovery, (B) mechanical work, (C) constraining stress.

In Figure 3 (B), useful mechanical work can be obtained since the stress developed in reversion is greater than the stress required for deformation. In this application (investigated by Cross et al, Ref. 2), the original shape is lost but an equilibrium position is obtained between strain-hardening and stress-reversion.

In Figure 3 (C), NiTi is used as a structural member. In this case it is a coupling in a piping system. In this application, the ease of attachment is the greatest benefit. The object is permitted to regain only a small portion of its initial shape. The advantage is the stress developed internally due to the constrained transformation process. It is the situation relating to the constrained application that is investigated in this study.

II. AREAS OF STUDY

The purpose of this study was to initiate investigations into the thermo-mechanical behavior of NiTi. Successful use of NiTi requires a broad data base to optimize design and fabrication parameters. The primary goal was to initiate an empirical study of the reversion of deformation martensite while constrained and thereby provide a data base for the engineering use of NiTi. Most earlier studies of the mechanical behavior of NiTi had investigated its elasticity, yield and flow stress. The test procedures required that temperature be held constant while straining the specimen. In most cases, the strain rate used was greater than 0.020 in/min.

In Goodyear's NASA research program, reported in Cross et al (Ref. 2), investigations were made of the tensile "recovery" stress, in which case the initial strain was held fixed. Using this procedure, the optimal value of initial strain to produce the maximum reversion stress was determined. Beyond this value of initial strain, the final value of reversion stress decreases from the maximum possible. The study provided engineering design data for a continuously constrained application of NiTi such as a direct conversion heat-engine. Not considered in the study was the use of NiTi where a portion of the initial strain would be lost.

Of specific interest in the present investigation was the general application wherein a NiTi member applies a constraining force to another member as a result of reverse martensitic transformation to austenite. In such applications the resultant stress is dependent upon both the partial loss of initial strain and temperature fluctuations following the reversion.

This study emcompassed four areas of interest:
1. the effect of partial reversion (of the initial strain) on the reversion stress; 2. the effect of applied stress on the temperature range for austenitic transformation; 3. the effect of cooling below M_d on the retained stress; 4. the stability of the retained stress.

Past investigations into the above or similar areas are briefly reviewed. Questions of interest to this study

which previously have gone uninvestigated are discussed. A statement regarding the general approach and an explanation of the goal conclude each section.

A. THE EFFECT OF PARTIAL REVERSION (OF THE INITIAL STRAIN) ON THE REVERSION STRESS

In most early research in NiTi martensitic reversion, specimens were allowed to revert unconstrained. Shape memory effect, as an identifiable characteristic, was defined. "Recovery" force (internal stress induced by the transformation from deformation martensite to austenite) was investigated by Cross et al (Ref. 2). The reversion stress developed by the transformation of deformation martensite was examined over a broad range of initial strains.

Not yet investigated was the problem of using this reversion stress of NiTi as an applicator or constraining force where-in the original shape is not recovered. A study of this nature appeared to be necessary if NiTi were to be used for loading applications such as in couplings or any other constraining mode (i.e. rivets, joint rings, turbine wheels). In these or similar applications, loss of initial strain due to tolerance requirements and compliance of the constrained member must be considered.

A dilatometric investigation of partial reversion was attempted. Starting from a predetermined initial strain, a portion of the initial deformation martensite was reverted by heating to thermal austenite. This event would occur

prior to arrest of the specimen by the constraining device. The objective of the investigation was to determine the effect of partial reversion of initial strain on the final reversion stress.

B. THE EFFECT OF APPLIED STRESS ON THE TEMPERATURE RANGE FOR THE AUSTENITIC TRANSFORMATION

A commonly accepted characteristic of NiTi is the narrow temperature range over which the martensitic and austenitic thermal transformation occur, as discussed in Wasilewski (Ref. 3). It is also commonly accepted that deformation martensite can be formed by an applied stress in certain temperature ranges. Dilatometric studies have been conducted to analyze the anelasticity of deformation martensite above the A_f. These studies have tentatively defined a limiting temperature, M_d, for the formation of deformation martensite. Figure 2 shows the general relationship between A_f and M_d on a relative temperature scale. The concept of starting with initial deformation martensite under stress and approaching A_f from a lower temperature has not been investigated.

A dilatometric investigation of the martensitic reversion to austenite was therefore attempted under rather special conditions. The goal was to observe the effects of externally applied stress, reversion stress, and partial loss of initial strain on the formation of austenite.

C. THE EFFECT OF COOLING BELOW M_d ON THE RETAINED STRESS

Past studies have investigated the occurrence of deformation martensite. As a result of these tests into the superelastic behavior of NiTi, a limit temperature, M_d (normally above A_f), was proposed as the temperature above which no deformation martensite may be formed, Wasilewski (Ref. 3).

In further studies, Cross et al (Ref. 2), M_d was identified by producing deformed martensite from stable austenite. Test specimens were cooled to various temperatures below an annealing temperature of 300°C. The specimens were then strained at constant temperature to determine the yield stress and modulus of elasticity. The results of these tests added to the validity of the M_d limit concept. While these data provided useful information for many applications, further insight was required for other situations.

The present study attempted to investigate a special application of NiTi. In particular, it was considered of interest to observe the reduction in stress of the constrained austenite due to the formation of deformation martensite as the temperature was reduced below the M_d (in a specimen previously reverted from deformation martensite to the state of constrained austenite). Although these conditions bear a close relation to the studies of Cross et al, there are sufficient variations to warrant study.

D. THE STABILITY OF THE RETAINED STRESS

No stress relaxation investigations of constrained austenitic or austenitic-martensitic NiTi have been reported. Schwenk, in Grumman's report on Rivet Fasteners (Ref. 4), reports the results of creep tests conducted at 65°C. A full investigation of this area was not considered within the scope of this study.

The present study attempted to directly observe changes in the retained stress at temperatures well above the A_f. The goal was to determine the stability of the retained stress and thereby the permanency of austenite formed from constrained deformation martensite.

III. EXPERIMENTAL PROCEDURES

All mechanical tests were performed in tension. The two factors guiding this decision were: one, the convenience of an underslung rig-configuration for rapid interchange of specimen environment; and, two, the sponsor's desire to use material in a tensile application. Mechanical tests were concluded on a 1000-lb capacity Instron testing machine, model TMS-L.

A. ALLOY COMPOSITION AND PROCESSING

The NiTi alloy used for testing was obtained courtesy of the Raychem Corporation. The alloy composition by weight was 49.18% Titanium, 49.82% Nickel, and 1% Aluminum. The alloy was prepared by electron-beam melting into an

ingot of 1.25-in diameter. The ingot was hot swaged to approximately 0.5 in. The bar was then cold drawn with a pass schedule of 10% - 15% diameter reduction per pass to final diameter of 0.0968 in $\pm$ .0002 in. As a final step the alloy rods were vacuum annealed at 900°C for thirty minutes and furnace-cooled, thus removing all previous strain history.

B. APPARATUS FOR MECHANICAL TESTING

1. Specimen Geometry

All specimens used were nominally one inch in gage length. Gage length for testing was defined to be the distance between the grips. Gage length was measured after mounting of the specimen in the grip assembly. The limit of accuracy of this measurement was found to be 0.0001 in. The sample alloy rods were cut into 1.7 in specimens using a carborundum cut-off wheel.

The specimen geometry and, subsequently, the rig design were chosen to take advantage of carefully prepared material supplied by Raychem. By configuring the test apparatus to accommodate a wire sample, no machining or subsequent vacuum annealing of the specimens was required.

The specimen was gripped in a vice-like manner. Figure 4 compares a specimen before and after testing. The center of the grips were drilled and tapped to provide a serrated gripping surface. The grips were then slotted to permit constriction of the specimen when the thumbscrews of the collars were tightened as shown in Figure 5. As a

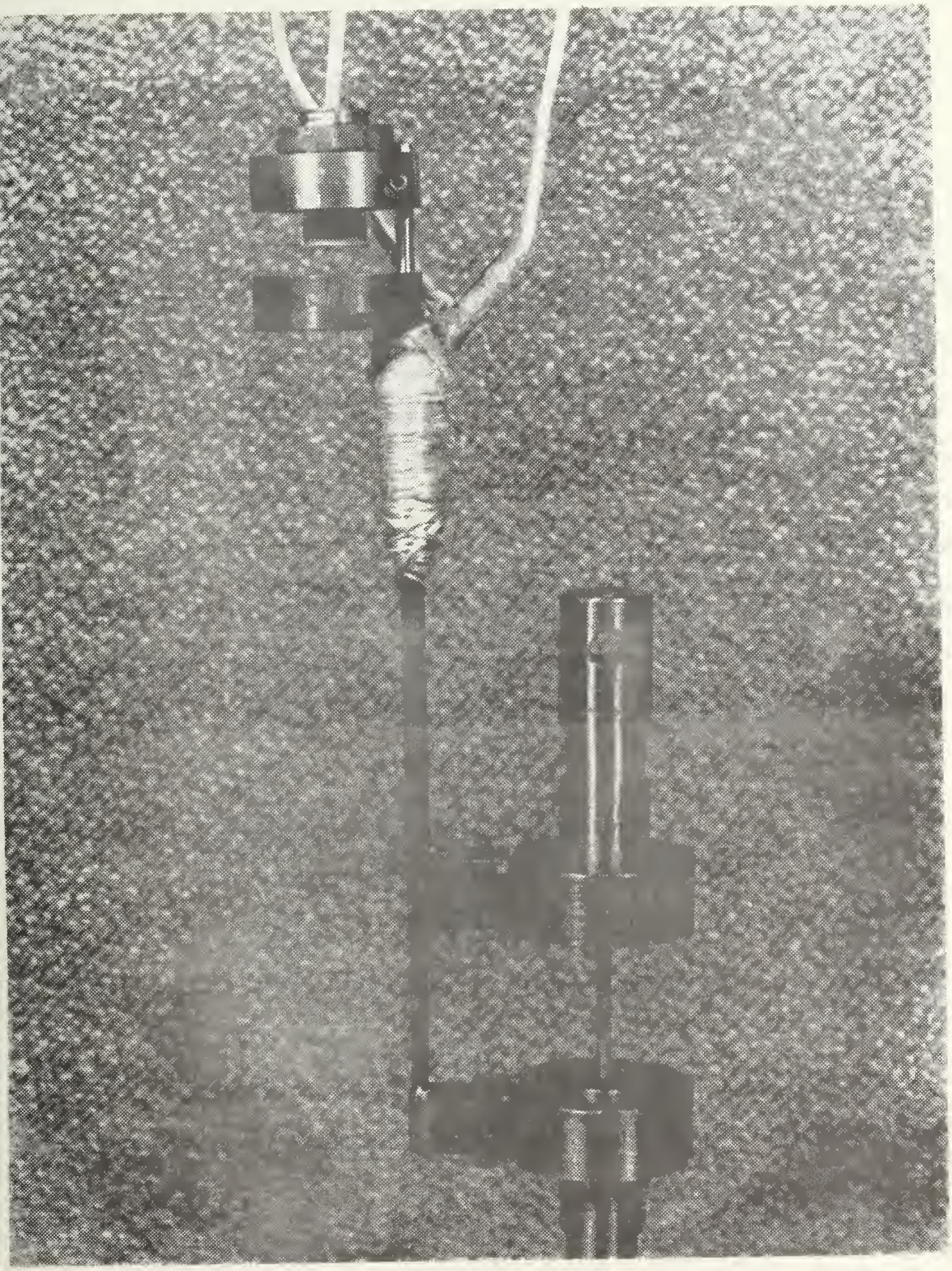


FIGURE 4. The serrated grip assembly with the proximeter extensometer mounted.

check on slippage and on the accuracy of gage length, additional gage marks were placed on the specimen. The marks were measured with a linear-measuring microscope with a repeatable accuracy of 0.0005 in.

2. Experimental Contrivance

The testing rig, fabricated from stainless steel, was designed to allow rapid interchangeability of immersion baths. An anvil, supported in compression by three columns, acted as an extension of the crosshead. The load cell was coupled to the upper grip by a reach rod. The reach rod projected through a center hole in the crosshead as shown in Figure 6, left.

The underside of the anvil and the base of the lower grip had matched spherical surfaces to permit self alignment. The reach rod and the upper grip were pin-jointed, and the anvil was slotted to permit installation of the grip assembly into the rig. See Figure 6, right.

3. Thermal Environment Control

Five separate thermal environments were used during the testing: free-boiling liquid nitrogen at -196°C, ethanol at -65°C and -50°C, water from 10°C to 100°C, silicon oil from 100°C to 250°C, and potassium nitrate from 350°C to 450°C. Temperature control was maintained in the silicon oil and the potassium nitrate by a Honeywell-Brown controller (0 - 1200°C) with an accuracy of $\pm$ 3°C. In the ethanol bath and the water bath, the temperature was controlled manually with similar accuracy. In all cases, with the exception of

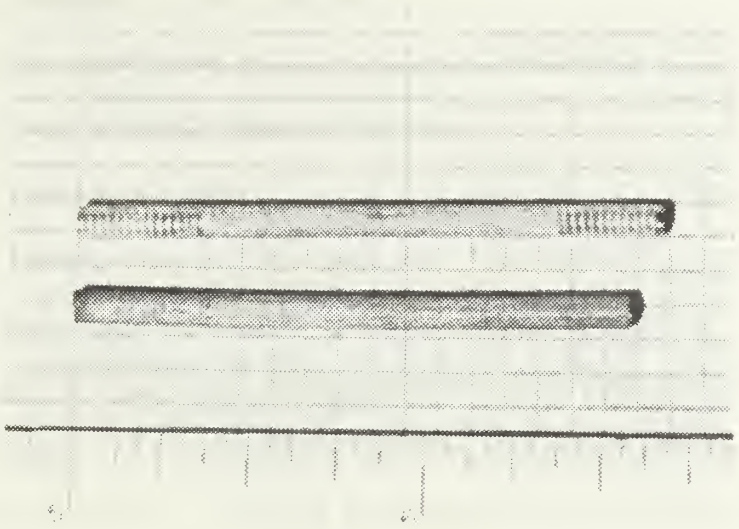


FIGURE 5. The NiTi tensile specimens before and after testing.

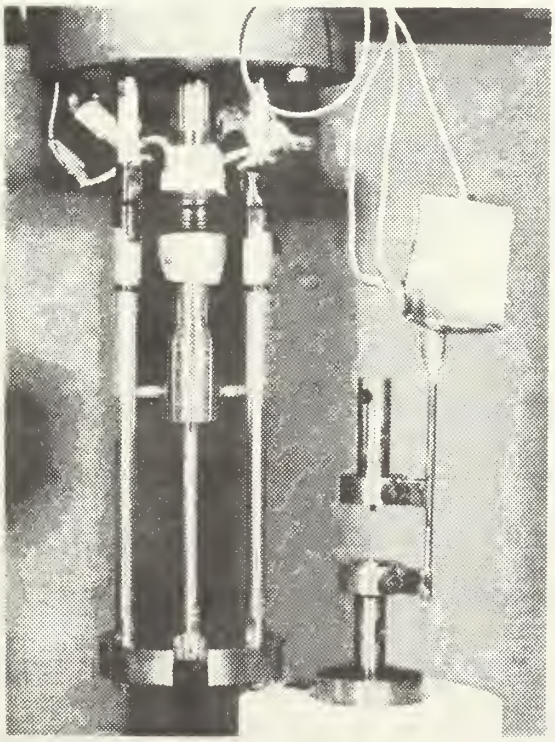


FIGURE 6. The testing rig with grip assembly and specimen ready to be installed.

liquid nitrogen, true temperature was determined using a Leeds and Northrop millivolt-potentiometer coupled to a Chromel vs Alumel thermocouple.

Heaters were placed on the rig to shorten the time to reach thermal equilibrium at deformation temperatures. A heater was also placed on the support posts for the proximeter transducer to keep this device in its thermally linear range. (See Figure 6 for arrangement.) A thermal-insulation jacket, fitted for forced-air cooling when necessary, was also provided for protection of the proximeter transducer.

4. Data Recording

Data were recorded from three sources. The Instron stripchart recorder provided load versus crosshead motion. A Hewlett-Packard stripchart recorder, model 7100B, coupled to the Bentley-Nevada proximeter, model 30B-L18, recorded engineering strain at the grips versus time. Where temperature was a controllable variable, the event marker on the Instron recorder was used to mark temperatures.

Varying thermal-gradients and the corresponding expansion and contraction of the rig inhibited the use of the crosshead position to determine engineering strain. In this capacity the proximeter, due to its compactness and accuracy, was an invaluable experimental tool. Crosshead position was used as a check of the proximeter operation. By assuming that volume change of the specimen was due only to the coefficient of thermal expansion, the data was reduced to true tensile stress versus true strain.

5. Mechanical Stiffness and Thermal Response Characteristics of the Apparatus

The response of the proximeter transducer was calibrated and found to be generally linear over a 0.100-in range. The deviations from linearity were found to be repeatable and correction factors were applied.

The transducer signal drifted at low temperature. With the use of heating coils, a systematic error of 4% over-indication was maintained. Signal shift due to expansion or contraction of the support posts was also correctable.

The mechanical stiffness of the rig was determined at four temperatures over the 0-lb to 1000-lb load range. The temperatures used were -195.5°C, 100°C, 200°C, and 350°C. A compliance of 1.7×10^{-5} in/lb was maintained for these tests.

During all tests the crosshead was fixed prior to the martensite reversion to austenite. It was considered necessary to define the deflection response of the rig for the temperature patterns used. The immediate response of the rig to the thermal gradients was to extend the specimen an additional 0.002 in followed by a contraction of 0.001 in to 0.002 in depending on the immersion bath used.

C. PROCEDURES FOR THERMO-MECHANICAL TEST

Mechanical testing was conducted in four distinct areas due to the multi-phase phenomenon which was investigated. The temperature patterns employed distinguished the area of testing.

1. Single-Phase, Stress-Strain Behavior

The stress-strain testing of austenite and martensite was conducted at discrete temperatures. All tests were conducted at a 0.002-in strain rate.

2. Reversion: Free and Constrained

Reversion testing of deformation martensite was conducted to determine the limits of revertable strain and maximum reversion stress for the temperature pattern used. In the former case, the specimen grip was unpinned and stress-free reversion of the specimen was permitted. In the latter, the crosshead was stopped with all stress retained prior to reversion.

3. Reversion: Partially Constrained with Temperature Gradient Control

Further tests combined partial stress-free reversion and constrained stress-reversion. The specimens were constrained by separating the base of the lower grip and the anvil by a portion of the total strain. As the specimen reverted, it was arrested by the anvil and the reversion continued as increasing stress. A typical test would require the specimen and rig to reach thermal equilibrium in the low temperature bath. The specimen was then strained to approximately 5.5 percent total strain. The portion alloted to stress-free reversion was then provided by adjusting the crosshead. The low temperature bath was then exchanged for the high temperature bath, and specimen deflection and stress were recorded.

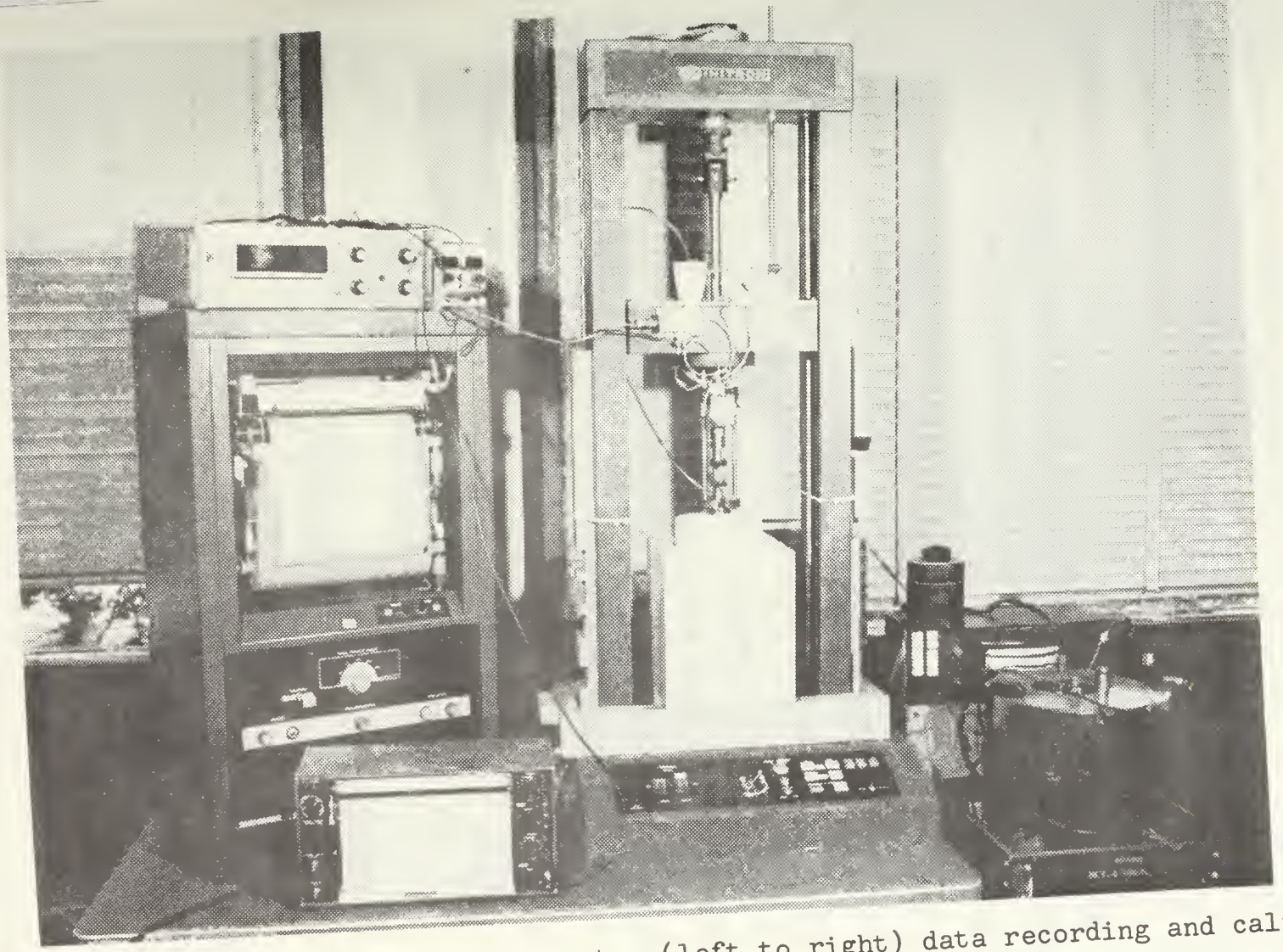


FIGURE 7. Mechanical testing equipment: (left to right) data recording and calibration, tensile testing, immersion bath and temperature controller.

A variation to this schedule was the use of an intermediate bath in which the temperature was gradually increased from 10°C to 100°C. This procedure permitted observation of the initiation and completion of austenite formation.

4. The Stability of Retained Stress in Austenite

Stress-strain data was obtained using specimens which had experienced constrained reversion to austenite. The test procedure required that the specimen be maintained at the final immersion-bath temperature for time periods up to 30 hours. The immersion-bath temperature was also held constant for each test at temperatures varying from 200°C to 377°C. During these tests both load and strain were recorded. These procedures permitted observation of the stability of the austenite formed by constrained reversion.

IV. RESULTS AND DISCUSSION

A. THE EFFECT OF PARTIAL REVERSION (OF THE INITIAL STRAIN) ON THE REVERSION STRESS

1. Results

The results of this portion of the investigation are plotted as two graphs with the same abscissa (see Figure 10). Above the abscissa is true stress versus temperature. Below the abscissa is true strain (starting at the initial value of true strain) versus temperature. The dotted line in each case indicates that the specimen was arrested and that reversion stress subsequently developed.

Included for comparison is the fully constrained case. Various fractions of initial strain were reverted prior to arrest. Additional loss of initial strain occurred after arrest due to the compliance of the experimental device.

Figure 11 indicates a gain of final reversion stress due to increases in the fraction of initial strain which was retained after the arrest of the specimen. The final reversion stress varies directly with the fraction of initial strain retained. The final stress value of the constrained case is plotted for comparison to the partial reversion cases.

2. Discussion

Figure 11 is a graph of the final stress value versus the fraction of initial strain retained. The final value of stress for the constrained case is 31 percent greater than the yield stress for annealed austenite at the same temperature. The specimen in the constrained case was constrained at the final value of flow stress while the -196°C bath was removed. As the specimen temperature increased toward A_s, the effective flow stress decreased causing additional strain (due to compliance in the rig). The external stress stabilized at 16,000 psi prior to reaching A_s. The reduction in flow stress due to straining at temperatures closer to M_f are shown in Figure 8 and has been confirmed by Rozner and Wasilewski (Ref. 5). Such was not the case with partial reversion where the removal of stress prior to increases in temperature and allowance for

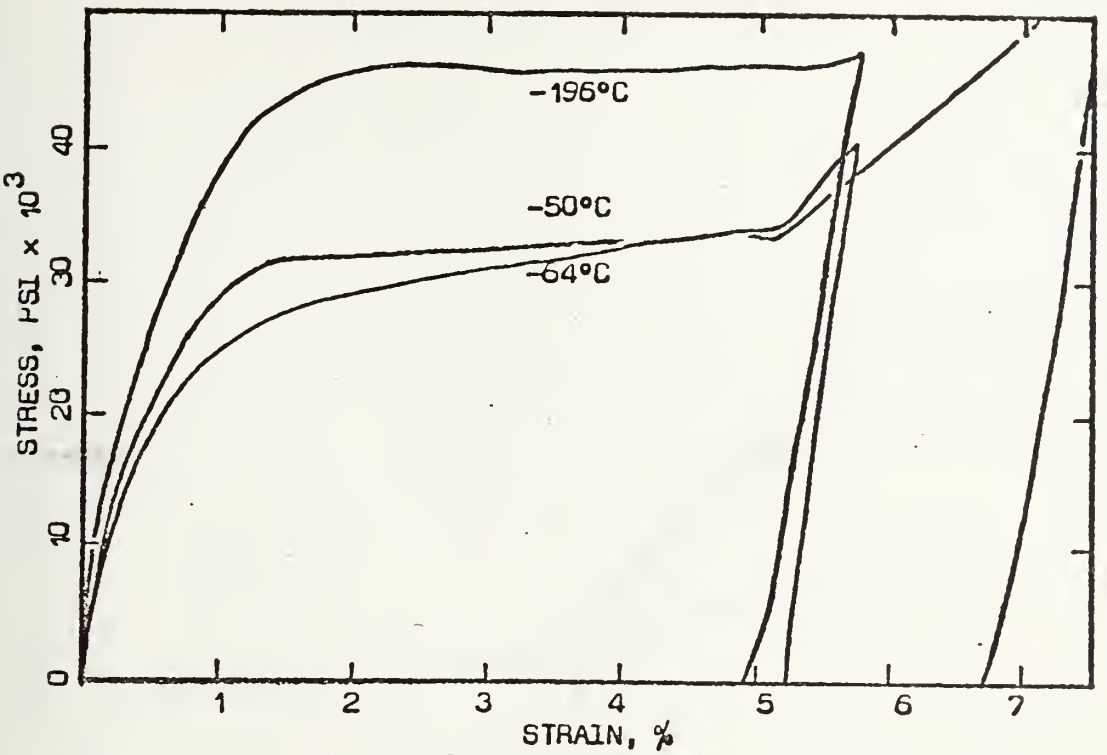


FIGURE 8. True stress vs true strain for martensitic NiTi at various temperatures below M_f.

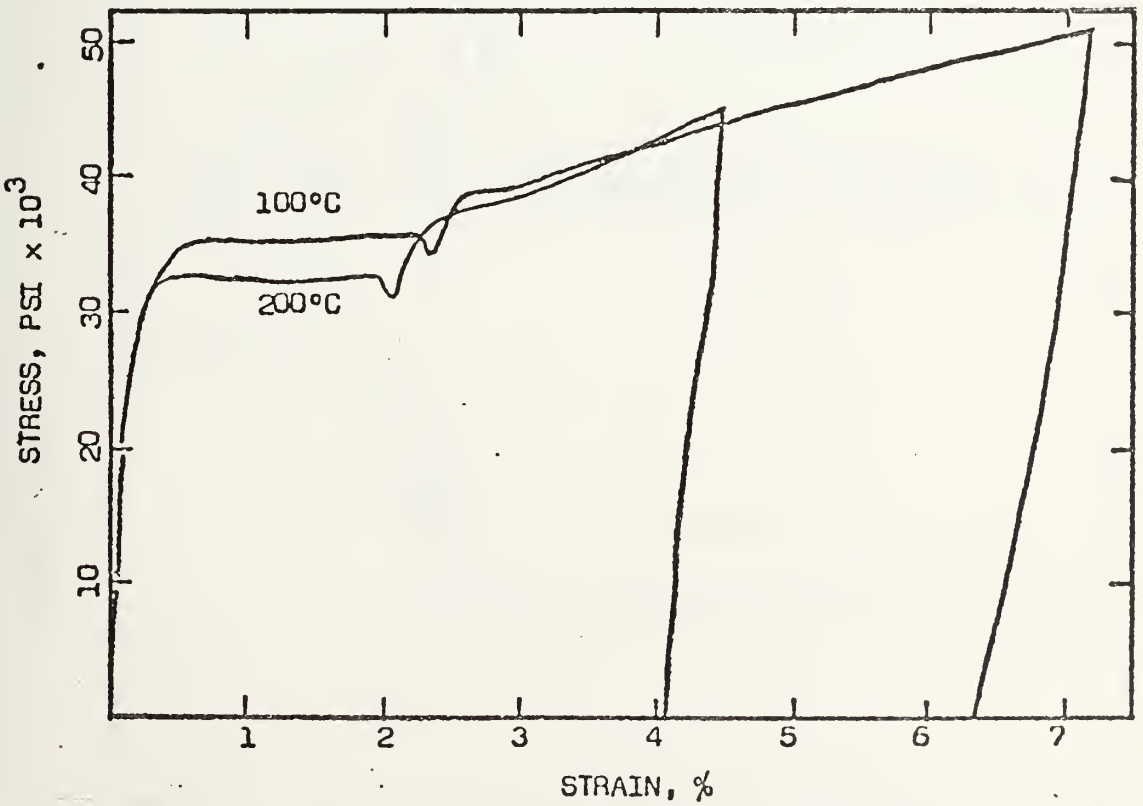


FIGURE 9. True stress vs true strain for austenitic NiTi at two temperatures above the M_d.

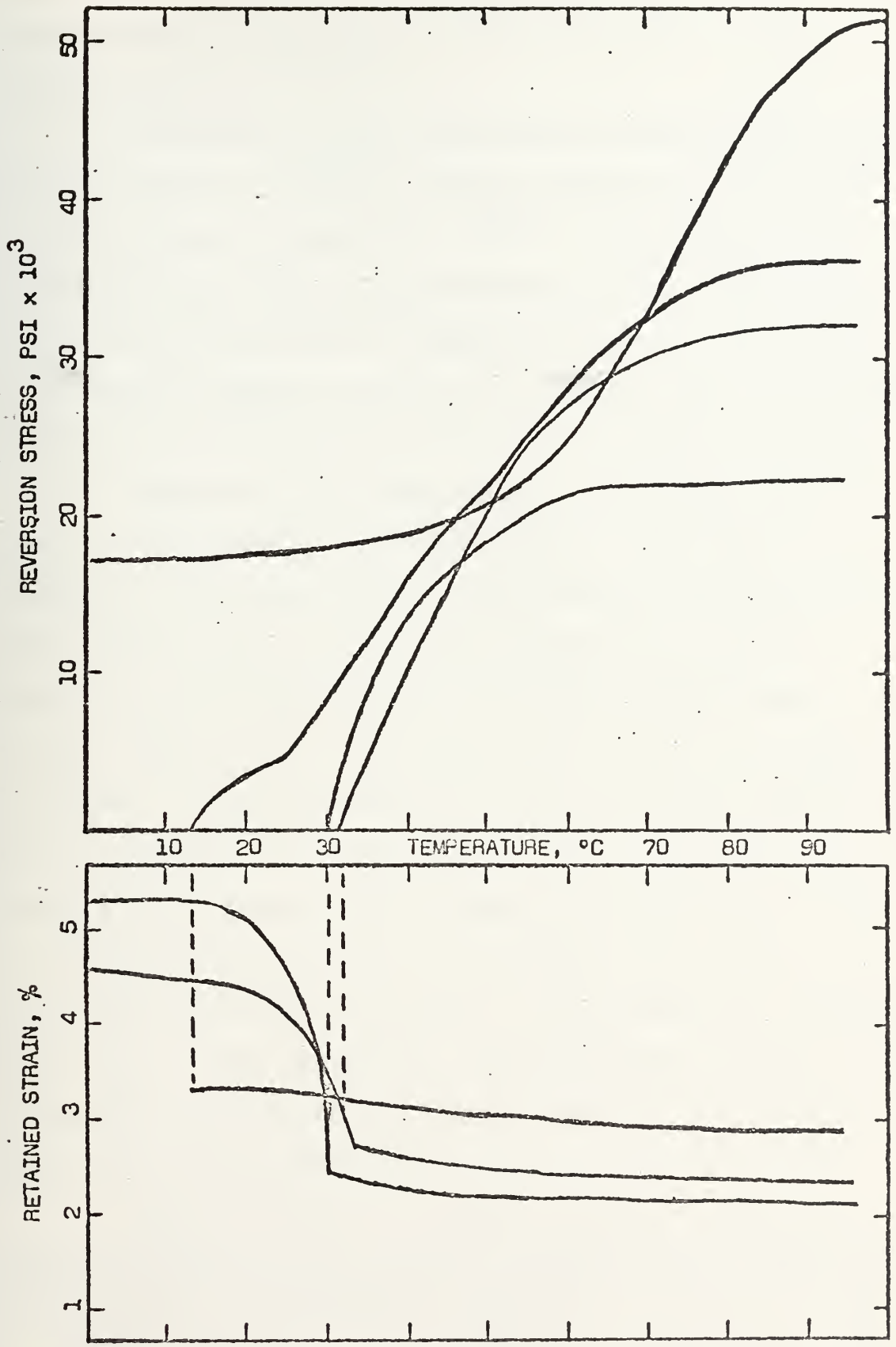


FIGURE 10. Comparison of the reversion stress for partial loss of initial strain. True stress and true strain vs specimen temperature.

strain reversion permitted a stress-free path for the initial reversion.

In Figure 11, the relationship between final stress and the fraction of strain retained appeared to be linear for the specimens strained at -196°C. In addition, the constrained case lies on the extension of that line. This correspondence indicates a dependence of the final stress on the strain retained and not the mode in which the reverted strain is lost (stress-free reversion or stress-reverted).

Contrary to the final stress values observed for specimens strained at -196°C are the two cases which were strained at -64°C and -50°C. The stresses developed for these cases at the corresponding fractions of retained strain are much higher than those of the initial tests. Although further tests are definitely in order, the indication is obvious. By increasing the initial straining temperature closer to the M_s, the final stress value for a given retained strain will increase. As an explanation for this behavior the straining conditions are considered. The stability of the thermal martensite increases as the temperature decreases (Ref. 6). It would therefore require greater stress to form deformation martensite from thermal martensite (Figure 8 indicates this correspondence).

In the process of forming deformation martensite at temperatures much lower than the transformation temperature, high densities of dislocation tangles are generated. Since these tangles are re-encountered in the reversion transformation back to austenite, a portion of the final stress

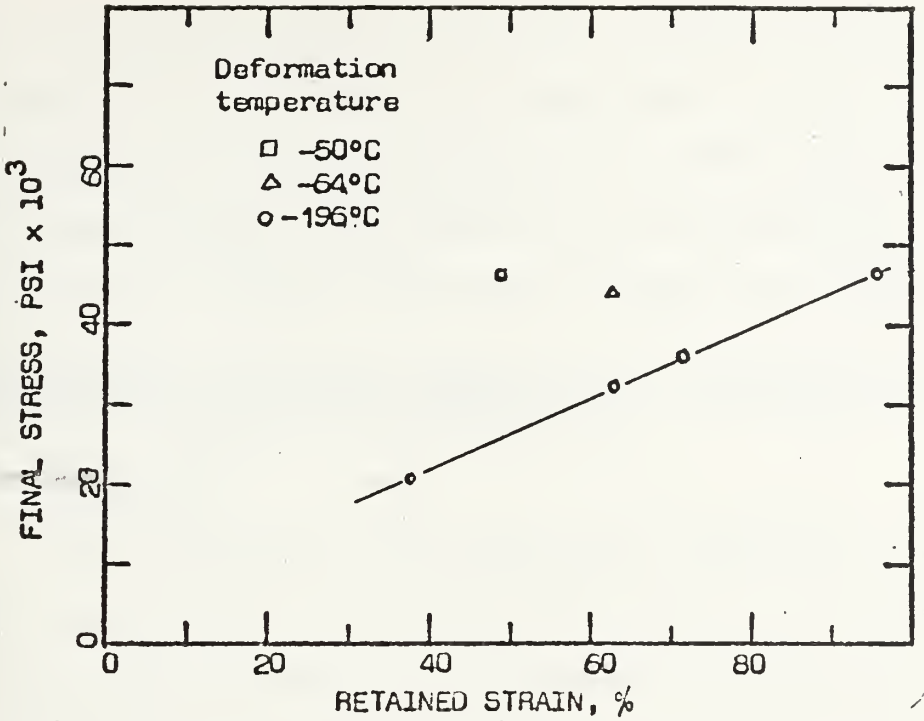


FIGURE 11. Increasing reversion stress due to greater strain retained in the specimen. Final true stress vs strain retained.

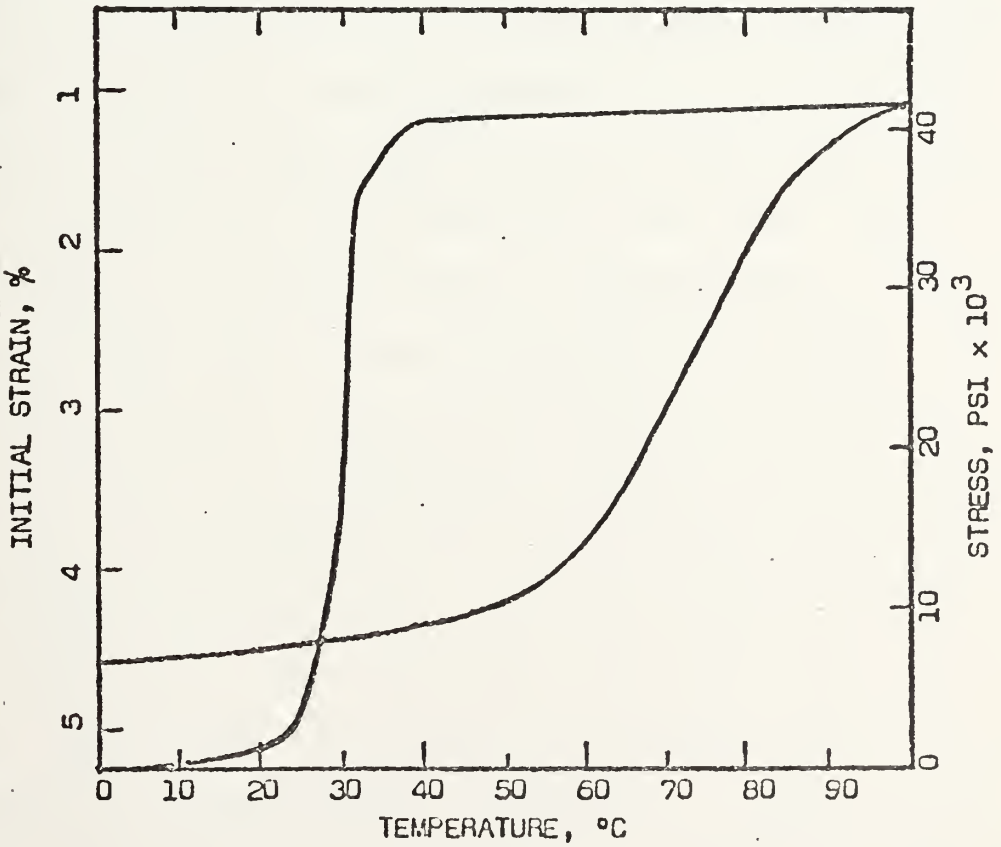


FIGURE 12. Comparison of the transformation temperatures for stress-free reversion and constrained stress reversion. Percent retained strain vs temperature to the left, and reversion stress vs temperature to the right.

capacity is lost to internal stresses. In the application under study, the end result is a lower final stress for a given fraction of retained strain. In addition, this behavior would explain the failure of the stress-free reversion case to regain its initial length (see Figure 12).

B. THE EFFECT OF APPLIED STRESS ON THE TEMPERATURE RANGE FOR THE AUSTENITIC TRANSFORMATION

1. Results

From Figure 10, the temperature dependence of the reversion process for the constrained case can be compared to the reversion process of the partially constrained cases. (Figure 12 compares stress-free reversion to stress-reversion.) All the partially reverted cases started the austenitic transformation in the stress-free reversion mode at approximately the same temperature. For the 35-percent, partially reverted case, the expansion of the experimental device interfered with the specimen's lower jaw. Minor stress was developed as thermal expansion continued but it was soon overcome at the start of the transformation process. This was confirmed by the temperature at which the increase in stress occurred. The temperature at which the transformations were completed increased as the final stress increased for the partially reverted cases. Also by comparing the partial reversion cases to the constrained case, this trend is continued. The results of the partial reversion cases as shown in Figure 11 are interpreted in Figure 13. Figure 13 has plotted the completion of austenitic transformation temperature,

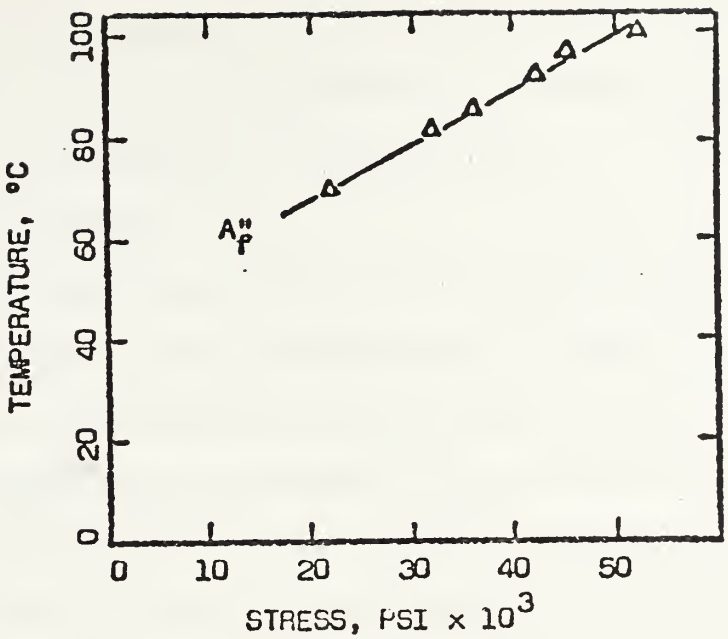


FIGURE 13. Increases in the transformation temperature due to increasing internal stress during constrained stress reversion. Temperature vs external stress.

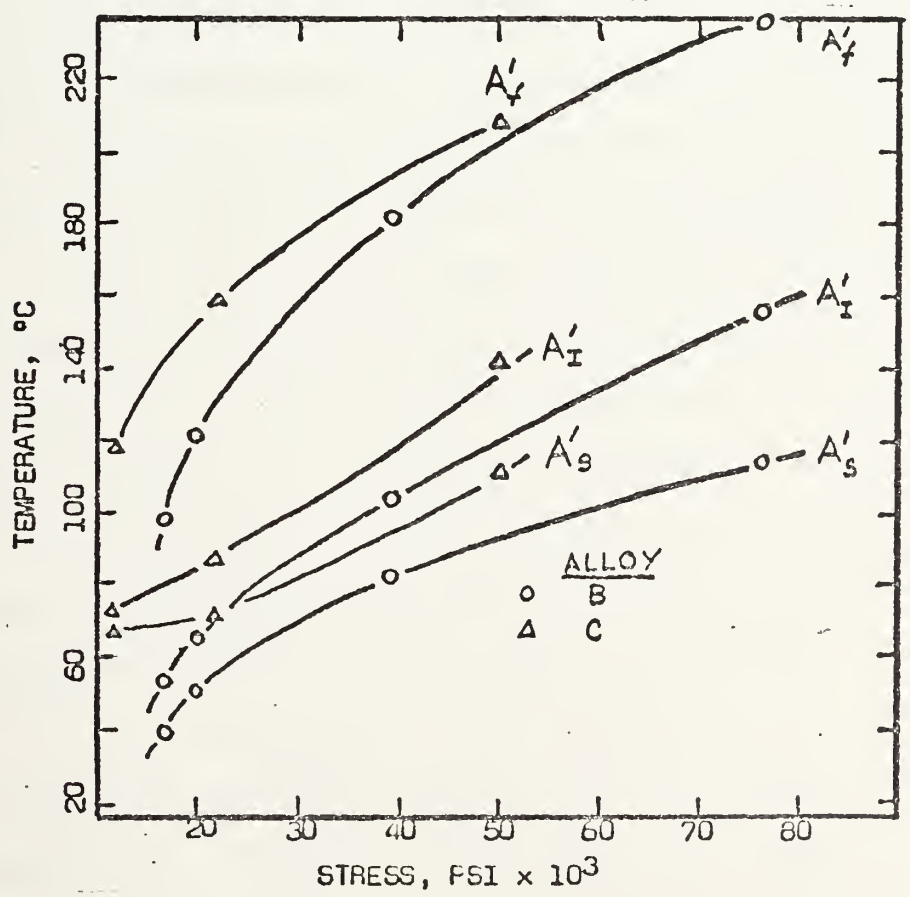


FIGURE 14. Increases in the transformation temperatures due to constant external stress during strain reversion. Temperature vs external stress (from data of Reference 2).

A''_f, under increasing reversion-stress. The prime signs indicate this variant case from the accepted terminology for austenitic transformation temperatures.

2. Discussion

The data indicated that the completion of the transformation process under stress was not solely dependent upon temperature. The external stress imposed upon the martensitic structure required an increase in the available energy to continue the transformation process. Schuerch, in Reference 7, reports an increase in transformation temperatures due to constant stress.

Similar results can be drawn from the "Mechanical Work" phase of Cross et al (Ref. 2). Figure 14 has plotted A'_s and A'_f temperatures for various degrees of initial strain and corresponding external stress. These data were gathered from Figures 15, 16 and 17 which themselves were taken from Reference 2. In these tests the load was constant and the specimens were allowed to recover some of the initial strain (part 3, p. 38 contains a more complete explanation of the test procedures). Since the stress was constant throughout, a single prime is used to indicate the variant case. Once again, because of the external stress, greater available energy was required to complete the transformation to austenite.

The increasing A''_f due to increasing developed stress is in agreement with the results of the mechanical work tests of Cross et al. The data points plotted in Figure 13 result from specimens deformed at three different temperatures and varying degrees of initial strain. Also included is one case

of constrained reversion. Apparent from Figure 13 is the linear dependence of A''_f on the developing stress.

Also of particular interest were the partial-reversion cases where the A_s was unchanged from the stress-reversion case but the A''_f was increased due to increasing internal stress. The transformation, once started, continued at a constant rate. The rate of reversion in one specimen (36-percent case) was slower than the constrained case. It is apparent from these results that the transformation process once started will continue with increasing temperature, regardless of self-applied or constant external stress. In tests in which the stress were artificially increased during reversion, it may be possible for the transformation to be retarded after it was started.

3. Observations on the "Mechanical Work" Tests in Cross et al

In Cross et al (Ref. 2), the ability of NiTi to perform mechanical work was investigated. The three alloys used in the study are titled A, B, and C. The compositions and characteristics of these alloys are listed in Table I. Rods with a 0.100-in diameter were strained uniaxially in tension by suspending a weight from the end of each rod. Varying degrees of initial strain were used in the tests depending on the mass of the suspended weight. The rods were initially strained at two different temperatures. The phase compositions at the straining temperature were not the same for all. Alloys A and B were composed of thermally stable β_o, but B was closer in temperature to its M_s. Alloy

C was composed of β_o and M_o in transition from M_s to M_f. Subsequent to the initial strain, the rods were heated while constant force was applied, and reversion of the initial strain as a function of increasing temperature was observed. When the strain reversion process was completed (signified by no change in the rod length) the specimens were then cooled to the initial straining temperature. Table II lists the initial strains, stresses and strain temperatures used. The strain-reversion data for these tests were plotted as specific work (force times the deflection per unit of material volume) versus temperature. Measurements of deflection were recorded for both heating and cooling. The results of these tests are shown in Figures 15, 16 and 17.

Each curve plotted has an identifiable temperature for the initiation and completion of the transformation from deformation martensite to austenite. Since the transformation is not that of thermal martensite to thermal austenite, the transformation symbols A_s and A_f do not identify the case. In lieu of an accepted standard, A'_s and A'_f have been chosen to indicate initiation and completion of the austenitic transformation under constant stress.

It was observed for each of these curves that there exists a definite inflection point in the strain-reversion process (with one exception). Such an inflection point does not exist in the thermal martensite to thermal austenite reversion process which generates a sigmoidal curve for austenitic composition versus temperation (Ref. 6). It also does not occur in the deformation martensite to austenite

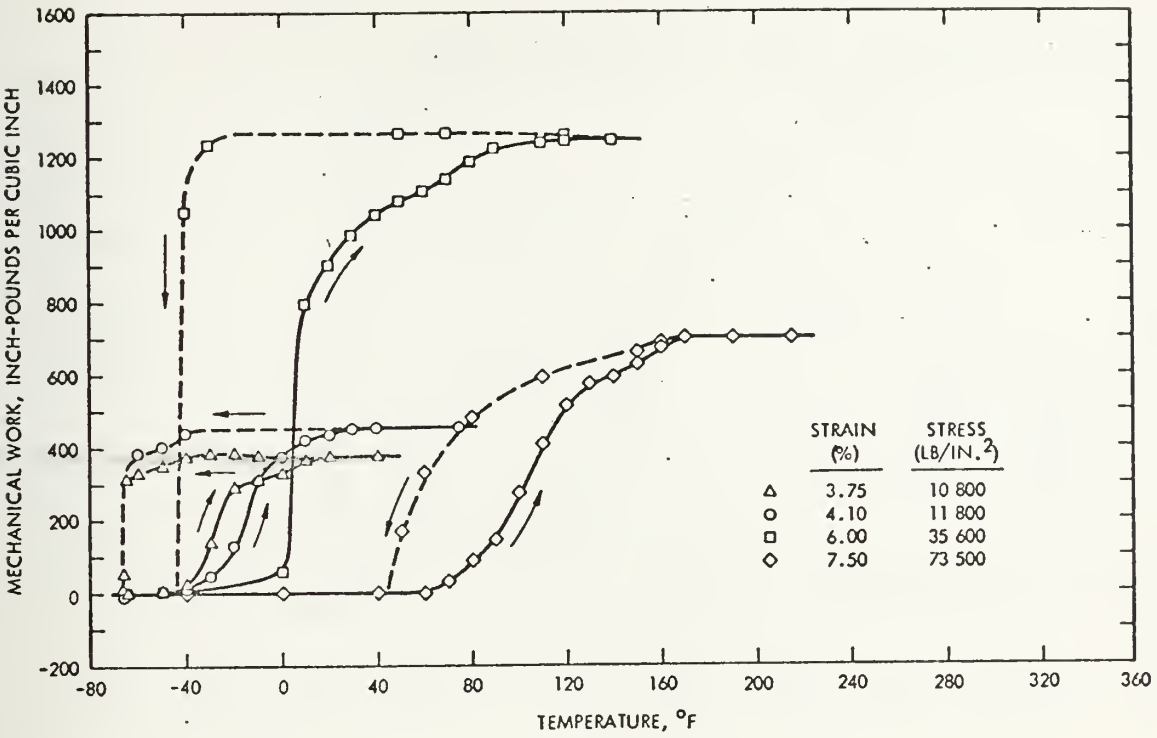


FIGURE 15. Mechanical work performed during strain reversion of alloy A. Specific work vs temperature.

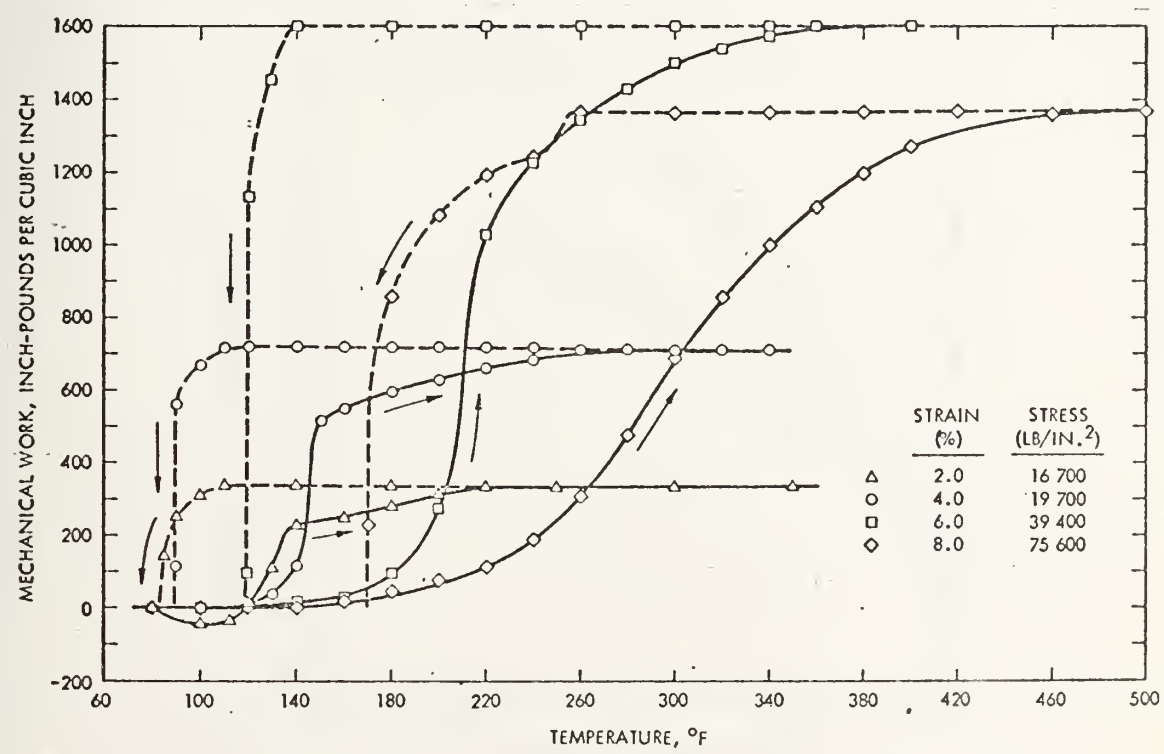


FIGURE 16. Mechanical work performed during strain reversion of alloy B. Specific work vs temperature.

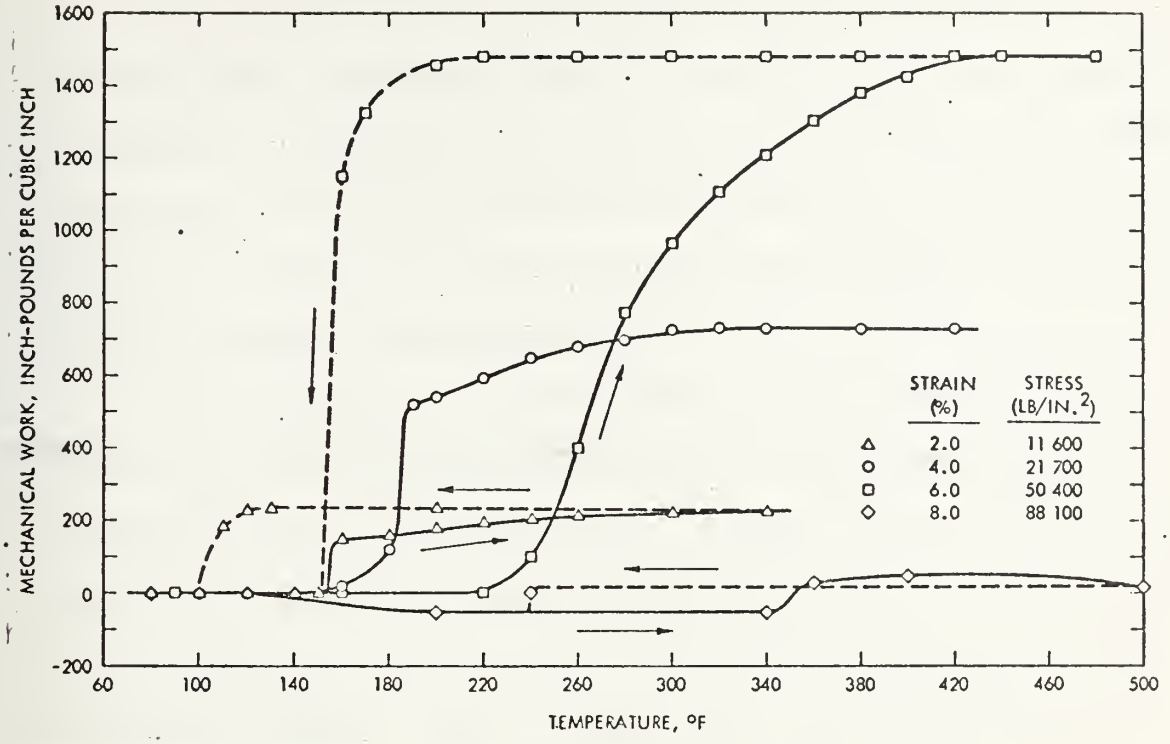


FIGURE 17. Mechanical work performed during strain reversion of alloy C. Specific work vs temperature.

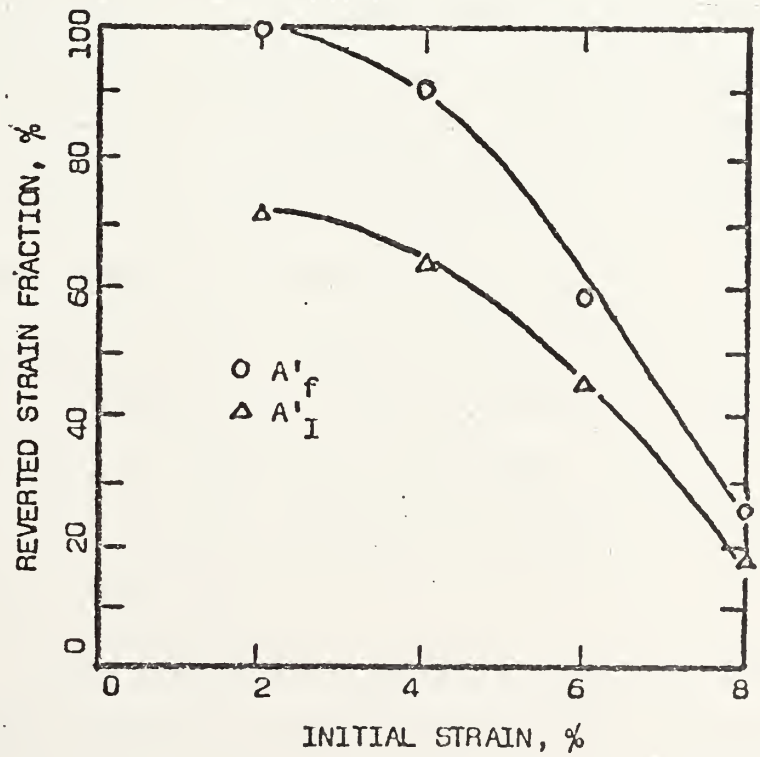


FIGURE 18. The fraction of the initial strain reverted at A'_I and A'_f vs the initial strain.

stress-free reversion of compression pre-strained NiTi which is also sigmoidal (Ref. 8). At the inflection point, a step-wise decrease in the first derivative of work versus temperature (rate of reversion) can be seen. Both above and below the inflection point, the rate of reversion is relatively constant with temperature.

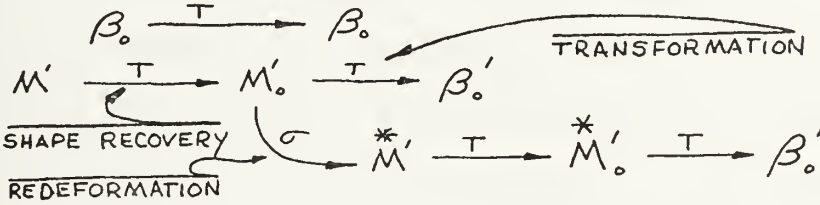
An attempt to characterize the occurrence of an inflection in the reversion process at some reproducible temperature, A'_I, was conducted. Figure 18 is a plot of the fraction of initial strain reverted versus the initial strain. Also plotted is the fraction of the reverted strain at the inflection point. The A'_I curve is proportional to the A'_f curve for the initial strains tested and also for varying amounts of constant stress. Inferred by this graph is the possibility that the A'_I is a function of the total amount of strain reverted. Figure 19 is a plot of the percentage of reverted strain at the inflection point ($A'_s \xrightarrow{\epsilon'_r} A'_I$ per the total reverted strain $A'_s \xrightarrow{\epsilon'_r} A'_f$). The fraction, ϵ'_r/ϵ'_T, is plotted relative to the initial strain. Curves are drawn for all three alloys. Again the A'_I appears to be proportional to the degree of reverted strain as seen by the horizontal best-fit curve for each alloy, and it is not dependent on the initial strain within the limits of the deformation martensite available.

As a possible explanation for the occurrence of an inflection point, the reversion process was considered to be the combination of two or more types of transformation from

deformation martensite to austenite. In alloys A and B, the initial phase was β_o . Under stress, the β_o transformed partially to M′.

$$\beta_o \xrightarrow{\sigma} M' + \beta_o \downarrow \qquad **$$

For this argument, it was assumed that deformation martensite, M′, formed from β_o , has differing reversion properties from deformation marteniste, $\overset{*}{M}'$, formed from thermal martensite. As the temperature is increased, the transformation to austenite produces metastable inter-phases.

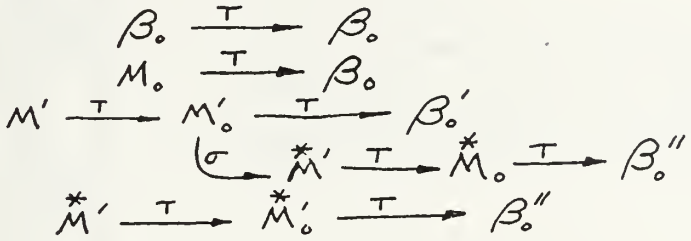


The transformation process from M′ to β_o' must first strain revert to M_o' prior to transformation to β_o' . Due to the applied stress, some of the M_o' is susceptible to deformation to $\overset{*}{M}'$. By the proposed model the $\overset{*}{M}'$ inter-phase transforms in a manner similar to the $M' \rightarrow M_o' \rightarrow \beta_o'$ transformation but requires a greater activation energy for transformation. In alloy C, the initial state was a combination of β_o and M_o. Under stress, this initial state transformed to two types of deformation martensite.

$$\beta_o \xrightarrow{\sigma} M' + \beta_o \downarrow$$
$$M_o \xrightarrow{\sigma} \overset{*}{M}' + M_o \downarrow$$

**Arrows indicate decreasing or increasing amounts of the phase in the transformation.

As with alloys A and B, the transformation introduces similar inter-phase products.



In this case, more of the $\overset{*}{M}'$ inter-phase is present.

This proposed model agrees with the observed behavior. As shown in Figure 19, the amount of strain occurring after A'_I increases as straining temperature decreases towards M_s with alloy C having the greatest degree of post-inflection strain. This observation is consistant with a greater fraction of $\overset{*}{M}'$ in the transformation process for the proposed model. Figure 20 proposes the interaction of these differing reversion rates to produce a characteristic inflection point.

C. THE EFFECT OF COOLING BELOW M_d ON THE RETAINED STRESS

1. Results

Figure 21 shows the results of two cases of cooling the reverted austenite while constrained. In both cases, the reduction in stress was accompanied by a 0.2 percent increase in strain. In case A, the specimen was stress-reverted to austenite with partial loss of initial strain. The final stress did not exceed the yield stress for annealed austenite. In case B, the specimen was stress-reverted while constrained. Since the specimen was externally stressed during transformation, no stress-free reversion was permitted.

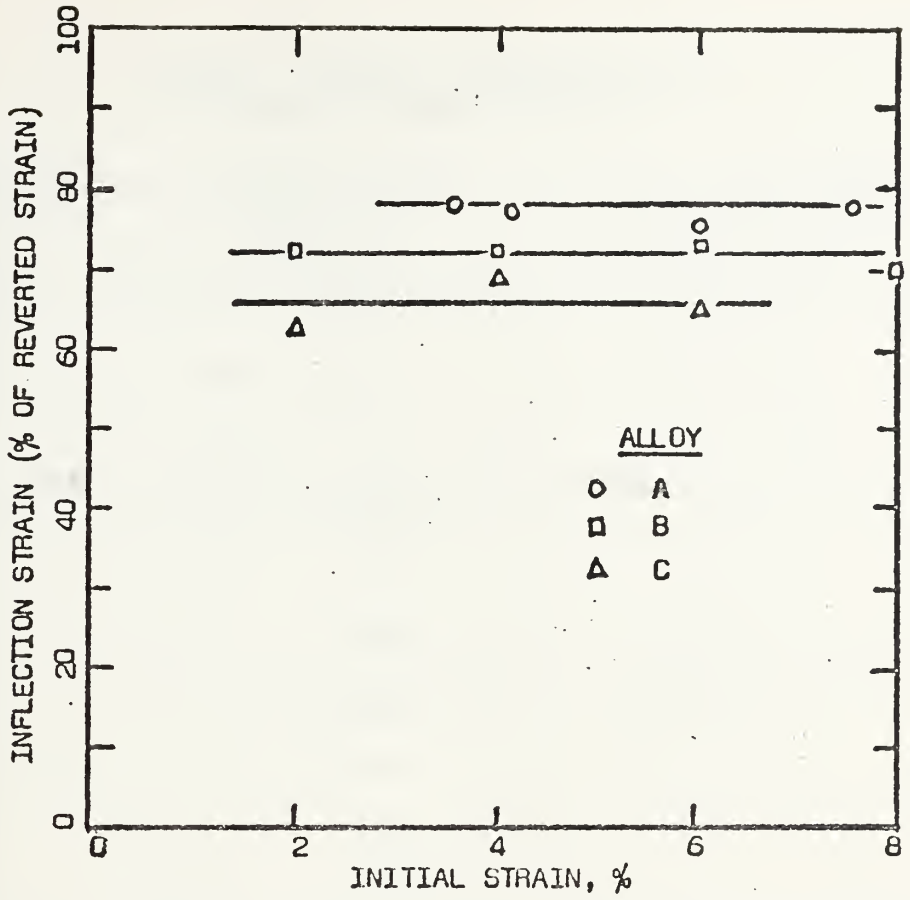


FIGURE 19. The occurrence of A'_I as a fraction of the total reverted strain in three alloys. Percent of reverted strain prior to the inflection vs the initial strain.

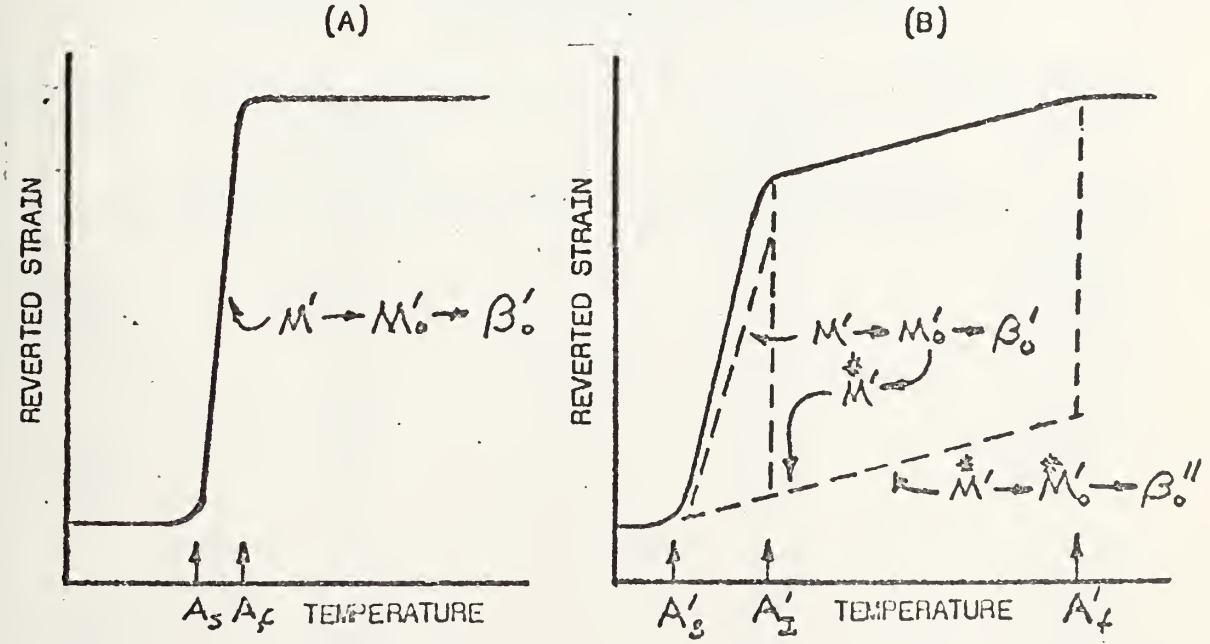


FIGURE 20. Schematic model of a reversion process (hypothetical): a. reverted stress-free, b. stress-reverted with an inflection point.

TABLE I ALLOY COMPOSITION AND THERMAL CHARACTERISTICS

ALLOY	COMPOSITION WEIGHT %	PHASE TRANSITION TEMPERATURES °C
A	Ni - 51.3 Ti - 42.2 Co - 6.5	M_d - 38 A_s - -51 M_s - -73
B	Ni - 55.00 Ti - 44.93 C - 0.07	M_d - 71 A_s - 43 M_s - 13
C	Ni - 54.54 Ti - 45.34 C - 0.06 S - 0.006	M_d - 88 A_s - 54 M_s - 32

TABLE II PARAMETERS FOR MECHANICAL WORK TESTING

ALLOY	INITIAL STRAIN IN/IN	FINAL STRAIN IN/IN	STRESS (PSI)
A STRAIN TEMP -54°C	3.75 4.1 6.0 7.5	3.5 3.9 2.0 1.7	10,800 11,800 35,600 73,500
B STRAIN TEMP 24°C	2.0 4.0 6.0 8.0	2.0 3.6 3.5 2.1	16,700 19,700 39,400 75,600
C STRAIN TEMP 24°C	2.0 4.0 6.0 8.0	1.9 3.3 3.0 0	11,600 21,700 50,400 88,100

The case-A specimen was cooled below the A_s to approximately the M_s. The linear rate of the stress relaxation from 100°C to 45°C indicated the formation of deformation martensite. Tests of the rig without specimen had shown only a 10,000-psi change in stress over the 0°C to 100°C range. However, from 45°C to 20°C, there was an increase in the stress-relaxation rate which was followed by a slower rate similar to the first. Upon heating, the stress reversion process retraced its path almost completely.

The case-B specimen was cooled to the A_s. As in case A, there was a change of stress-relaxation rate at approximately 40°C which continued until the cooling was stopped. When heated, the stress reversion process assumed the same shape with no hysteresis effect. An 8 percent loss in the reversion-stress was observed.

2. Discussion

Case A was able to regain practically all of its original stress, whereas case B lost 8 percent. The difference was attributable to the lower initial stress in case A. In case A, the reversion stress did not exceed the yield stress of the austenite that was formed. In case B, two possible explanations for the loss of stress are proposed. One possibility is that insufficient deformation martensite was formed during the cooling to permit reversion to the original stress level. The alternate explanation involves the strain hardening of the austenite. The specimen was initially strain-hardened during the first stress-reversion

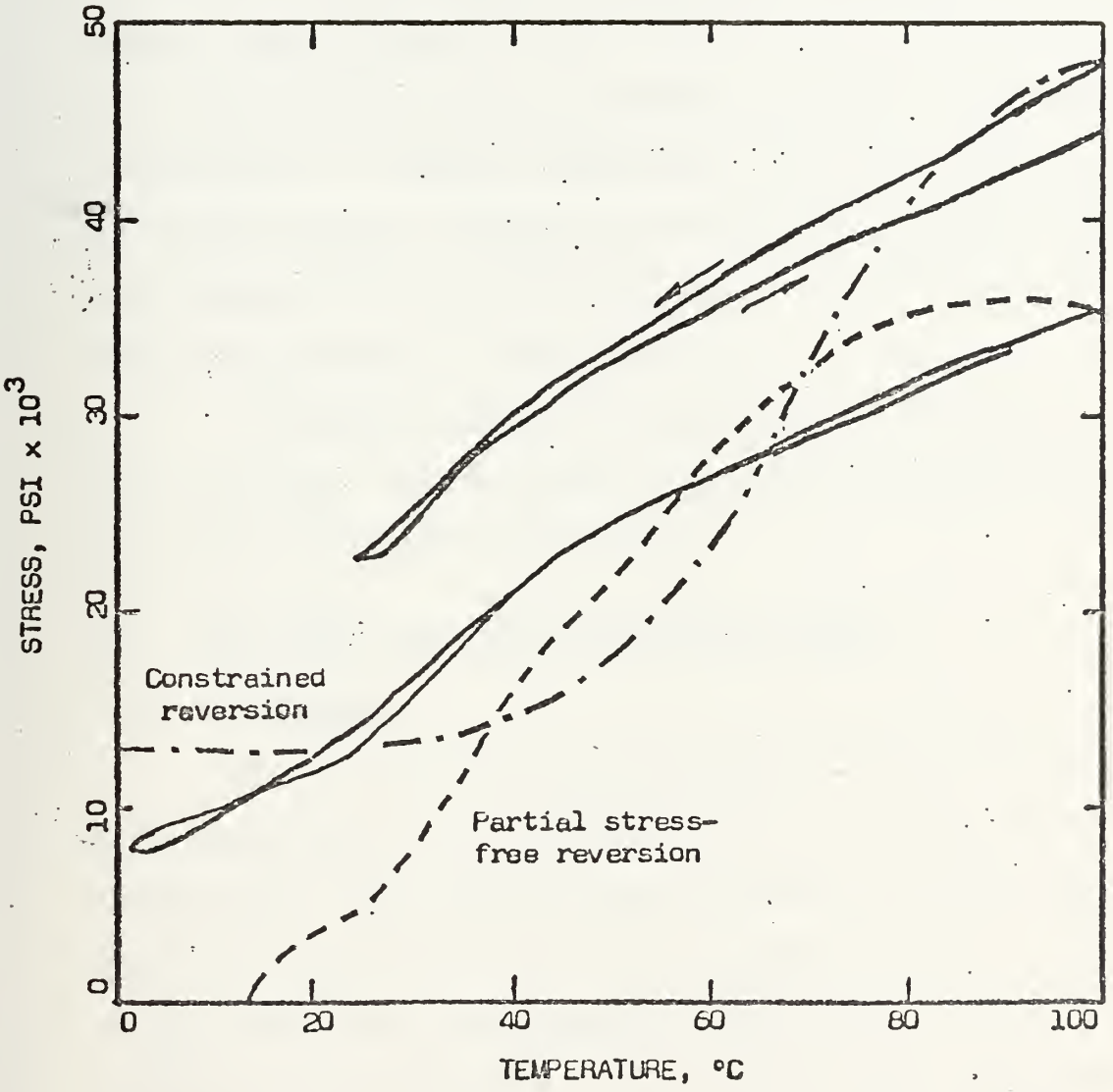


FIGURE 21. Reversion-stress recovery after cooling below the A_f. True stress vs true temperature.

process. When the specimen was stress-reverted for the second time, more strain-hardening occurred. The dislocation tangles created by the first strain-hardening inhibited the growth of deformation martensite during the cooling phase. Additionally, these tangles were encountered in the transformation back to stable austenite. By this explanation, a higher temperature would be needed to complete the stress reversion to austenite. Similar conditions were encountered by Wasilewski while investigating the fatigue strength of NiTi (Ref. 6).

Further testing in this area is necessary before any conclusion can be drawn as to which process provides the more reasonable explanation.

D. THE STABILITY OF THE RETAINED STRESS

1. Results

The results of this portion of the investigation were inconclusive. As explained in the Experimental Procedures (p. 29), the specimens, after partial reversion, were held under the resultant reversion stress in the high-temperature baths for periods varying from 18 to 30 hours. Only in the constrained case was any stress relaxation observed. This occurred after 20 hours at 377°C, with a final stress value of 34,000 psi, which had relaxed from 36,000 psi.

Thermal expansion of the experimental device caused a reduction in the stress from 8,000 psi to 12,000 psi depending upon the immersion-bath temperature. In the

partial-reversion tests, this complication reduced the stress to a range from 12,000 psi to 15,000 psi. The time periods used may not have been sufficient under these low-stress conditions to display any stress-relaxation activity.

2. Discussion

Since no stress-relaxation parameters can be calculated from a single case, and since no prior stress-relaxation testing has been conducted, very little can be said.

As a final observation, the austenite formed by stress reversion is apparently a stable austenite and appears to behave as strain-hardened austenite. Rozner and Wasilewski (Ref. 5) reported that from 100°C to 400°C, the yield stress for annealed austenite was constant at 35,000 psi.

V. CONCLUSIONS

Conclusions drawn from this investigation are enumerated below:

1. The final value of reversion stress is linearly dependent on the fraction of retained strain.

2. The final value of reversion stress decreases as the temperature for the initial formation of deformation martensite decreases.

3. The final value of reversion stress is not dependent on the mode of lost strain (either stress or stress-free reversion).

4. A_s and A_f increased with greater values of developed stress during the reversion. Also the A_s and A_f temperature shift is independent of the amount of initial strain or the temperature of initial strain.

5. In the case of partial stress-free reversion the transformation starts at the free reversion temperature and continues smoothly regardless of the developing stress to the new A_f.

6. Observed strain-reversion kinetics (under constant stress) are consistant with a proposed duplex reversion model.

7. The final value of reversion stress can be regained after cooling to temperatures below the M_d. Reductions in the stress regained are experienced when the final reversion stress is sufficient to strain harden the formed austenite.

8. The reversion stress developed by partial and constrained reversion is stable when held at temperatures above the A_f.

VI. SUMMARY DISCUSSION AND RECOMMENDATIONS

In this section the conclusions that have been drawn from the data and results and their significance to engineering applications will be discussed. Recommendations for further investigations will be made where appropriate.

The results of section IV. A. indicate that the reversion transformation of deformation martensite, with initial loss of strain, can generate strain hardened austenite. Also indicated is a direct correspondence between the fraction of

retained strain and the final stress. An additional conclusion of engineering significance is the indication that increases in the initial deformation temperature (yet remaining below M_f) can increase the final stress for a given fraction of retained strain.

Further studies in this area are recommended. Comparisons of the final stress vs retained strain curves for various initial deformation temperatures would provide valuable engineering data. In addition, the comparison of initial deformation yield and flow stress to the final stress values would illuminate preferred straining temperatures.

Section IV. B. displayed the stress dependency of the transformation temperature, A''_f. This dependency appears to be linear with increasing stress. Also the A''_f shift is apparently independent of the degree of initial deformation or the temperature of the initial deformation. The effect of this behavior on local stress risers in an engineering application must be considered.

Further study in this area is warranted. More testing with greater control on the variable parameters will better define the dependence of the A''_f. In addition, reversion tests of traditional geometric stress risers would provide guidance for engineering design.

In section IV. C. it was determined that the final reversion stress can be regained subsequent to cooling below the M_d. The regained stress is diminished when the austenite formed by reversion transformation is strain hardened.

The change in strain during this cycle is limited solely to the compliance of the constraining device. Further tests in the strain hardening mode will better define the repeatability of the regained stress due to many temperature cycles.

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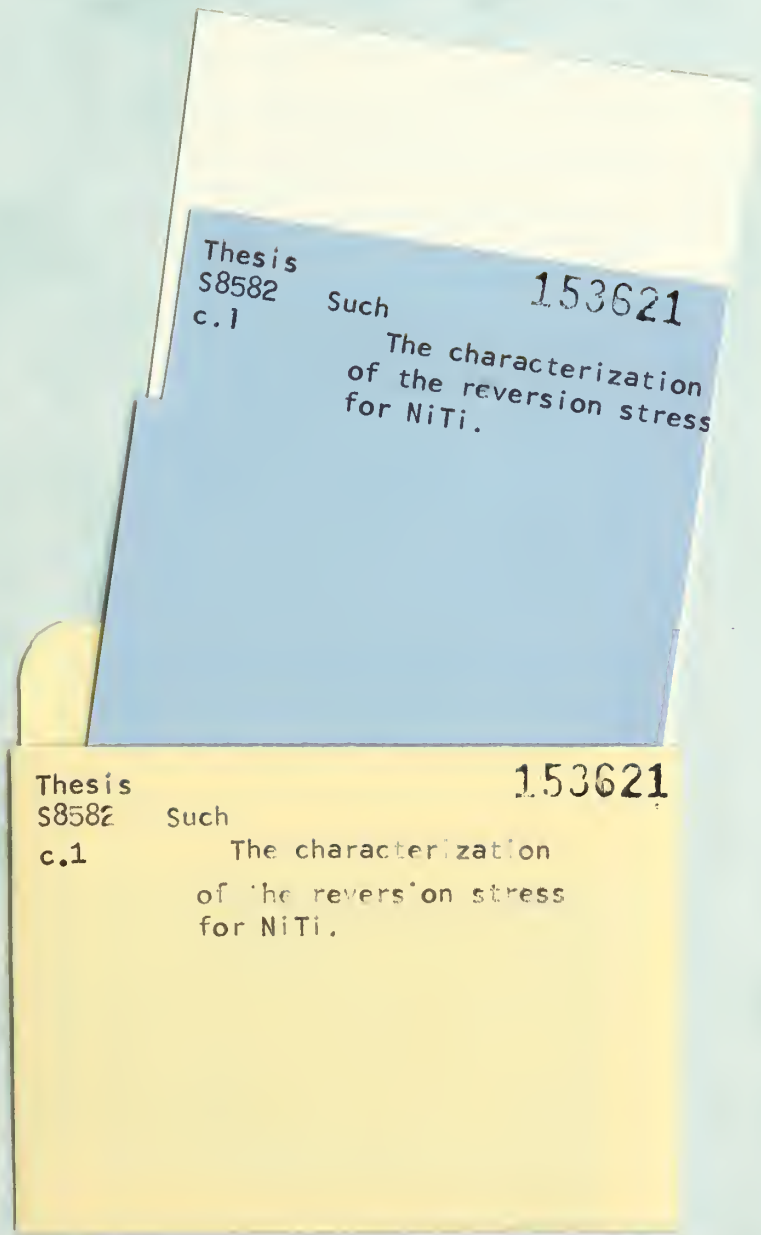
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